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**ELECTRON MICROSCOPE TECHNIQUE
SUGGESTED TO REVEAL MICROSTRUCTURES
OF DISPERSION-STRENGTHENED MATERIALS**

by Bruno C. Buzek

Lewis Research Center

Cleveland, Ohio

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NATIONAL AERONAUTICS AND SPACE ADMINISTRATION

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ELECTRON MICROSCOPE TECHNIQUE SUGGESTED TO REVEAL MICROSTRUCTURES OF DISPERSION-STRENGTHENED MATERIALS

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SUMMARY

This report describes a preparation and replication method which has produced consistently good results for a number of materials studied at the NASA Lewis Research Center. To show the effectiveness of the method evolved, measurements of volume percentages of dispersoids were compared with values obtained from chemical analysis. Typical microstructures of differently produced dispersion-strengthened materials are shown.

The method that was found to be satisfactory uses conventional metallographic specimen preparation procedures up to the two final steps of polishing. For this final polishing, 3-micron and 1/2-micron diamond polishing compounds are necessary. This produces a flat and distortion-free surface. Etching of the samples should be extremely light. The etchants used depend on sample composition and history. The samples are then cleaned in an ultrasonic bath and further cleaned with plastic by dry stripping. Replication of samples with a conventional two-stage method of plastic-carbon and platinum shadowing follows. A good correlation was obtained between volume percent dispersoid measured on the resulting electron micrographs and the values obtained by chemical analysis.

This replication procedure was found satisfactory for use with nickel, tungsten, chromium, cobalt, and their alloys. With suitable etchant changes, this replication procedure could also be applied to other dispersion-strengthened metals.

INTRODUCTION

During the past few years considerable effort has been expended by NASA at the Lewis Research Center to develop electron microscopy techniques that will yield the true microstructures of dispersion-strengthened materials. Such microstructures must clearly reveal dispersoid morphology and distribution so that the particle-statistics

mathematics described by Cremens (ref. 1), Fullman (ref. 2), and others can be applied quantitatively to their analysis. Of particular concern at this time for high-temperature use are nickel-base alloys containing ultrafine, uniform, stable dispersions of thorium oxide particles that are 0.05 micron, or less, in size, with interparticle spacings of the order of 1 micron.

Perhaps the most important procedure involved in electron microscopy of this type of material is the production of satisfactory replicas of the surfaces of the product. Although there are many possible methods for preparing, etching, and replicating specimens and individual microscopists might prefer to use approaches of their own, at times it is desirable to have a method which will produce results suitable as base-line data for comparison. It is the intent of this report to describe and suggest a method that has produced consistently good results for a variety of materials studied at the NASA Lewis Research Center.

The replication procedure to be described has been found satisfactory for use with nickel, tungsten, chromium, cobalt, and their alloys. With suitable etchant changes, this procedure also should be satisfactory for use with other dispersion-strengthened metals.

The following steps comprise the preparation procedure for replication:

- (1) Mounting
- (2) Polishing with 3- and 1/2-micron diamond compound
- (3) Etching
- (4) Ultrasonic cleaning
- (5) Strip cleaning with replicating medium
- (6) Replication
- (7) Shadowing and carbon deposition
- (8) Dissolution of support plastic

MOUNTING AND POLISHING

Specimens to be studied normally should be mounted in a plastic to facilitate handling. The samples may then be metallographically polished by using any conventional procedure. However, the final two steps of polishing should be conducted with 3-micron and 1/2-micron diamond polishing compounds, successively. These last two polishing steps quickly produce a flat and distortion-free surface with minimum tendency for microconstituents to pull out.

ETCHING

The etchant to be used will obviously depend on sample composition and, while one or two etchants usually are superior to others, any of a number may be satisfactory (table I). Our experience has indicated that for most nickel and nickel-base materials the preferred etchant is one generally called Ni-etch (92 ml hydrochloric acid, 3 ml nitric acid, and 5 ml sulfuric acid) diluted 1:4 with water.

As a general rule, the etch applied to dispersion-strengthened materials should be extremely light, that is, etching time is about one-fourth the time generally used in light microscopy.

CLEANING

After etching, the sample is submersed in an ultrasonic bath of alcohol for 1 minute and dried in a warm air blast. The sample surface is further cleaned by being dry-stripped two or three times as follows: Two or three drops of 0.25 percent Mowital dissolved in chloroform are applied; this covers the surface. The sample is tilted as soon as the Mowital touches it, and all excess Mowital (which will run to the edge) is removed by blotting with filter paper. The Mowital layer is then backed with 1.5 percent Parlodion in amyl acetate. The two plastic layers are dry-stripped with pressure-sensitive cellulose tape. This removes surface debris not removed by ultrasonic cleaning and renders the surface ready for replication.

REPLICATION

The sample is replicated and backed as outlined previously for dry-stripping (i. e. , Mowital plus Parlodion plus pressure-sensitive cellulose tape). The stripped replica is shadowed with platinum by using platinum pellets in the hollow end of one carbon electrode. A layer of carbon about 0.01 micron thick is then deposited vertically.

Next, the entire replica is cut into grid-size squares which are placed in amyl acetate to dissolve the Parlodion and remove the pressure sensitive cellulose tape (which floats away). The Mowital (replicating material) which does not dissolve in amyl acetate is kept intact and used in combination with the platinum-shadowed carbon layer as the replica. When the dispersoids in the replica are very small, the resolution may be enhanced if the Mowital film is completely dissolved in chloroform, leaving only the shadowed carbon layer. This platinum-shadowed carbon replica will then float free and can be collected on grids for subsequent electron microscopic examination.

Platinum is used as the shadowing material, and carbon is used as the supporting film. The carbon gives an essentially structureless film, has strength, and is thin enough to yield the best obtainable resolution to detect dispersoids that are 0.01 micron, or smaller, in size. Furthermore, the carbon conducts heat induced by the electron beam away from the initial replicating material and thus reduces the possibility of damage during electron microscope observation.

In order to obtain the best possible resolution and most accurate surface topography, overshadowing and overetching should be avoided. Likewise, in selection of the replica areas to be photographed, the purpose of the study must be considered, whether specific exceptional structures or representative structures are sought.

Typical replica electron micrographs of dispersion-strengthened materials are shown in figures 1 to 3. The nickel - 2-volume-percent-thorium oxide material (TD-Nickel) is one of the many exceptions to table I, in that an electrolytic cyanide etch produced somewhat better results than did the nickel etch. Figures 2 and 3 show micrographs of a nickel-base material and an aluminum-base material for which etchants from table I were used.

An indication of a successful application of the specimen preparation and typical replication techniques described herein is shown in table II (data from ref. 3). It represents typical results from measurements of volume fraction dispersoid made on replica micrographs of TD-Nickel and of aluminum - 5-volume-percent-aluminum oxide material (SAP M257). The quantity of oxide obtained from chemical analysis compares very well with that measured by using the techniques of this report. Equally good results were obtained regardless whether the oxide particles were discrete spheres or aggregated platelets.

CONCLUDING REMARKS

The method for preparing replicas for electron microscopy presented in this report has proved to be successful in revealing surfaces of such materials as dispersion-strengthened and conventional alloys. A comparison of measurement of quantities of dispersoids in a metal with quantities obtained from chemical analysis showed good agreement. Experience over a period of several years has indicated that measurements made on replicas prepared by the method of this report could be correlated with optical microscopy and chemical analysis.

It should be emphasized that this technique is just one of several which could be

applied to obtain replicas, but it has produced consistently good results at the NASA Lewis Research Center.

Lewis Research Center,
National Aeronautics and Space Administration,
Cleveland, Ohio, January 17, 1968,
129-03-01-05-22.

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2. Fullman, R. L.: Measurement of Particle Sizes in Opaque Bodies. Trans. AIME, vol. 197, no. 3, Mar. 1953, pp. 447-452.
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TABLE I. - SATISFACTORY ETCHANTS FOR VARIOUS MATERIALS

Material to be etched	Composition of etchant	Method of applying etchant
Aluminum - dispersoid	2 g potassium hydroxide - 1 g potassium chloride - 100 ml water	Swab
Cobalt - dispersoid	3 percent bromine in methanol	Swab
Chromium - dispersoid	10 percent sulfuric acid in water	Electrolytic at 3 V
Copper - dispersoid	10 ml ammonium hydroxide - 2 ml hydrogen peroxide (30 percent)	Swab or dip
Nickel - dispersoid	92 ml hydrochloric acid - 3 ml nitric acid - 5 ml sulfuric acid (diluted with water 1:4)	Swab
Nickel-molybdenum-tungsten - dispersoid	20 ml hydrochloric acid - 15 ml ethyl alcohol - 15 ml water - 2.5 g copper chloride	Swab
Nickel-chromium alloys	10 ml hydrochloric acid - 5 ml nitric acid - 0.5 g copper chloride	Swab
Tantalum	20 ml hydrofluoric acid - 1 ml nitric acid	Swab
Tungsten	30 ml lactic acid - 10 ml nitric acid - 5 ml hydrofluoric acid	Swab

TABLE II. - VOLUME PERCENT OF DISPERSOID
OBTAINED BY CHEMICAL ANALYSIS AND BY
ELECTRON MICROGRAPH AREA ANALYSIS
IN TD-NICKEL AND SAP M257 MATERIALS

Sample material	Chemical analysis		Electron micrograph area analysis (a)
	Source A	Source B	
TD-Nickel	2.05	1.91	1.90
SAP M257	4.2	----	5.0

^aVolume percent = A/A_M , where A is total area of particles in field scanned, and A_M is area of electron micrograph scanned.



Figure 1. - Carbon replica of nickel - 2 volume percent thorium oxide electrolytically etched with 10 percent sodium cyanide in water at 10 volts dc. X30 000.

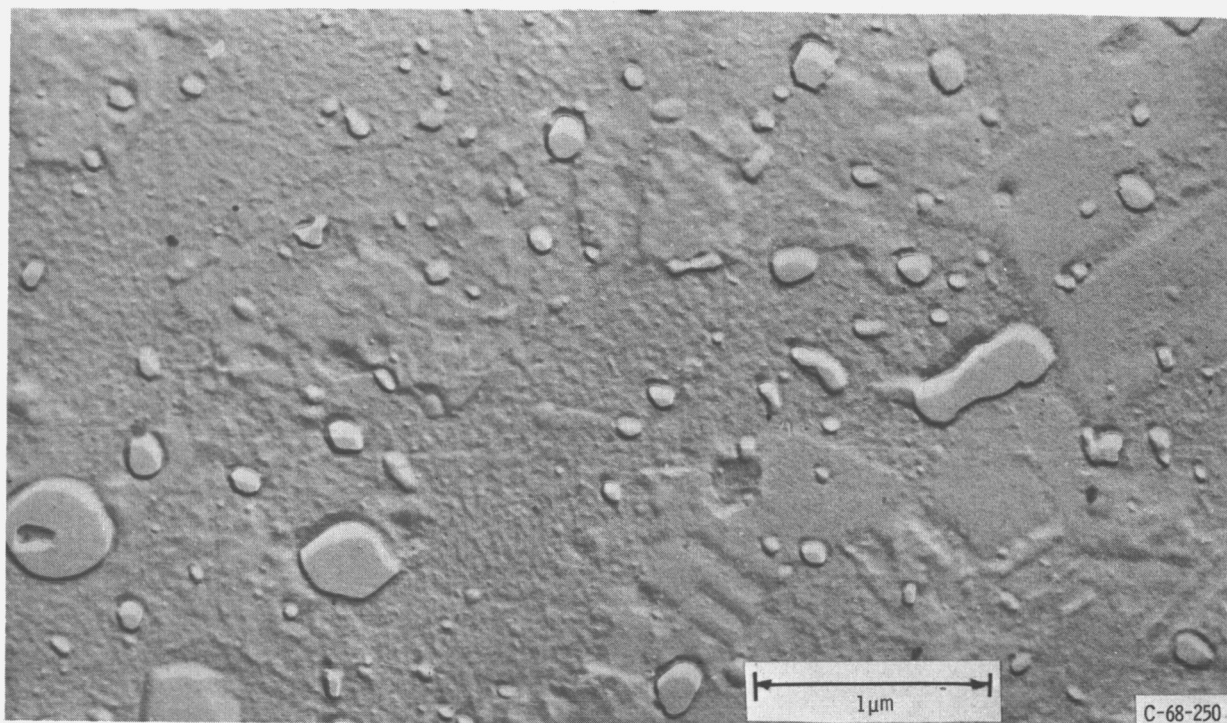


Figure 2. - Carbon replica of nickel - 8 volume percent thorium oxide. Nickel etch; X30 000.

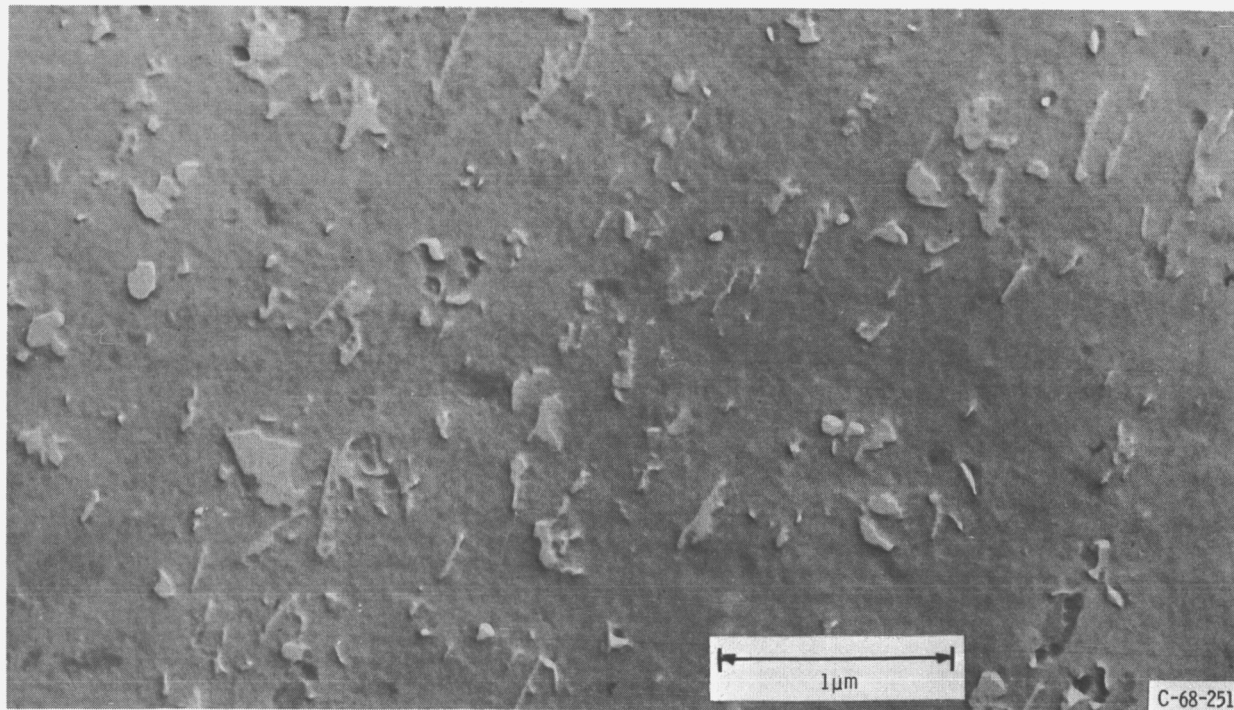


Figure 3. - Carbon replica of aluminum - 5 volume percent aluminum oxide. Etchant, 2 grams of potassium hydroxide and 1 gram of potassium chloride in 100 milliliters of water; X30 000.